

# Visualization of Domain Inverted Regions in Periodically Poled Lithium Niobate Using X-ray Microdiffraction

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## Introduction

Because of its high electrooptic and nonlinear optical coefficients, ferroelectric lithium niobate is commonly used in optical devices. An additional degree of freedom in optical design is gained by artificially manipulating domains of electrical polarization – for example in quasi phase-matching experiments. The real-space structure of polarization domains in LiNbO<sub>3</sub> and other ferroelectric materials can be exploited in applications ranging from electronic memories to advanced optoelectronic devices.<sup>1,2</sup> We show that the ferroelectric domain structure of a periodically poled lithium niobate (PPLN) crystal can be observed using x-ray microdiffraction.

## Method

In the absence of absorption, the beams produced by diffraction from crystalline reflections related by inverting the Miller indices (i.e.  $(hkl)$  and  $(\bar{h}\bar{k}\bar{l})$ ) have equal intensities. This principle is known as Friedel's Law. It was first shown in 1931, however, that anomalous scattering modifies the intensities of these reflections and that this effect can be used to determine the absolute direction of polarization of a crystal lacking inversion symmetry.<sup>3</sup> We have used this difference in intensity as the contrast mechanism in the quantitative mapping of the polarization domains in PPLN. Previous experiments along these lines have included x-ray topographic studies of domains in LiNbO<sub>3</sub> at the scale of several tens of microns.<sup>4</sup>

The x-ray microprobe beam was formed by focusing 10 keV incident photons from a two crystal Si (111) monochromator with a 12.5 cm focal length Fresnel zone plate. We selected radiation focused to the first order spot using a 20  $\mu\text{m}$  order-sorting aperture. The width of the beam at the focus was extracted by fitting an error function to step in the intensity of the Cr K $\alpha$  radiation from the edge of a 200 Å-thick Cr film scanned across them beam. Ambient vibrations limited smallest beam diameter to 2  $\mu\text{m}$  at the optimum focus. The sample was oriented using a kappa-geometry diffractometer and scanned beneath the incident beam using a servo-driven piezoelectric positioner. We used a silicon photodiode to measure the intensity of the diffraction. The 500  $\mu\text{m}$  thick PPLN sample was fabricated from a (001) oriented (z-cut) crystal.

## Results

An image formed by measuring the intensity of the  $\text{LiNbO}_3$  (006) reflection in a  $50 \times 50 \mu\text{m}^2$  area is shown in Figure 1(a). The intensity of the diffracted beam is modulated in a series of vertical stripes that reflect the series of domain inversions in PPLN. The mean wavelength of the domain inversions measured from the three domain periods intercepted by a linear scan along the dashed line of Figure 1(a), shown in Figure 1(b), is  $15.9 \mu\text{m}$ . This agrees with  $15.1 \mu\text{m}$  measured optically in a region 23 periods wide.

An estimate of the degree of contrast between domains can be made by including the effect of anomalous dispersion in calculations of the structure factors of the (006) and  $(00\bar{6})$  reflections. A calculation using the atomic coordinates of the ferroelectric phase of  $\text{LiNbO}_3$  and tabulated atomic scattering factors<sup>5,6</sup> predicts that the (006) reflection will be more intense than the  $(00\bar{6})$  reflection by a factor of 1.15, which is in reasonable agreement with the observed contrast  $I_{(006)}/I_{(00\bar{6})}=1.08$ . The sampling of an area of the sample outside the intense focus spot due to unfocused transmission through the zone plate is a possible contributor to the lower than expected experimentally observed contrast. A substantially larger difference in the intensities of these reflections can be expected when the beam energy is tuned to an atomic resonance (i.e. the Nb K edge near 19 keV). We plan future experiments to verify this prediction.

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Paul G. Evans, *et al.*

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Figure 1: (a) X-ray microprobe image of polarization inverted regions in  $\text{LiNbO}_3$  formed using the  $(006)$  and  $(00\bar{6})$  reflections. (b) A scan along the dashed on the image.

